high accuracy is required, the sample can be taken to dryness on a hot plate and the residue dissolved in the appropriate volume of water to bring it into the optimum range of the calibration curve.

11.4.2 Container No. 3 (Silica Gel). This step may be conducted in the field. Weigh the spent silica gel (or silica gel plus impinger) to the nearest 0.5 g; record this weight.

11.5 Check for Matrix Effects. Use the Method of Standard Additions as follows to check at least one sample from each source for matrix effects on the Pb results:

11.5.1 Add or spike an equal volume of standard solution to an aliquot of the sample solution.

11.5.2 Measure the absorbance of the resulting solution and the absorbance of an aliquot of unspiked sample.

11.5.3 Calculate the Pb concentration C_m in µg/ml of the sample solution using Equation 12–1 in Section 12.5.

Volume corrections will not be required if the solutions as analyzed have been made to the same final volume. Therefore, C_m and C_a represent Pb concentration before dilutions.

Method of Standard Additions procedures described on pages 9–4 and 9–5 of the section entitled ''General Information'' of the Perkin Elmer Corporation Atomic Absorption Spectrophotometry Manual, Number 303–0152 (Reference 1 in Section 17.0) may also be used. In any event, if the results of the Method of Standard Additions procedure used on the single source sample do not agree to within ±5 percent of the value obtained by the routine atomic absorption analysis, then reanalyze all samples from the source using the Method of Standard Additions procedure.

12.0 Data Analysis and Calculations

12.1 Nomenclature.

A_m = Absorbance of the sample solution.

A_n = Cross-sectional area of nozzle, m^2 (ft^2).

A_t = Absorbance of the spiked sample solution.

B_{ws} = Water in the gas stream, proportion by volume.

C_a = Lead concentration in standard solution, µg/ml.

C_m = Lead concentration in sample solution analyzed during check for matrix effects, µg/ml.

C_s = Lead concentration in stack gas, dry basis, converted to standard conditions, mg/dscm (gr/dscf).

I = Percent of isokinetic sampling.

L_1 = Individual leakage rate observed during the leak-check conducted prior to the first component change, m^3/min (ft^3/min)

L_a = Maximum acceptable leakage rate for either a pretest leak-check or for a leak-check following a component change; equal to 0.00057 m^3/min (0.020 cfm) or 4 percent of the average sampling rate, whichever is less.

L_i = Individual leakage rate observed during the leak-check conducted prior to the ''ith'' component change (i = 1, 2, 3 * * * n), m^3/min (cfm).

L_p = Leakage rate observed during the post-test leak-check, m^3/min (cfm).

m_t = Total weight of lead collected in the sample, µg.

M_w = Molecular weight of water, 18.0 g/g-mole (18.0 lb/lb-mole).

P_{bar} = Barometric pressure at the sampling site, mm Hg (in. Hg).

P_s = Absolute stack gas pressure, mm Hg (in. Hg).

P_{std} = Standard absolute pressure, 760 mm Hg (29.92 in. Hg).

R = Ideal gas constant, 0.06236 [(mm Hg) (m^3)]/[(°K) (g-mole)] {21.85 [(in. Hg) (ft^3)]/ [(°R) (lb-mole)]}.

T_m = Absolute average dry gas meter temperature (see Figure 5–3 of Method 5), °K (°R).

T_{std} = Standard absolute temperature, 293 °K (528 °R).

v_s = Stack gas velocity, m/sec (ft/sec).

V_m = Volume of gas sample as measured by the dry gas meter, dry basis, m^3 (ft^3).

$V_{m(std)}$ = Volume of gas sample as measured by the dry gas meter, corrected to standard conditions, m^3 (ft^3).

$V_{w(std)}$ = Volume of water vapor collected in the sampling train, corrected to standard conditions, m^3 (ft^3).

Y = Dry gas meter calibration factor.

ΔH = Average pressure differential across the orifice meter (see Figure 5–3 of Method 5), mm H_2O (in. H_2O).

θ = Total sampling time, min.

θ_1 = Sampling time interval, from the beginning of a run until the first component change, min.

θ_i = Sampling time interval, between two successive component changes, beginning with the interval between the first and second changes, min.

θ_p = Sampling time interval, from the final (n^{th}) component change until the end of the sampling run, min.

ρ_w = Density of water, 0.9982 g/ml (0.002201 lb/ml).

12.2 Average Dry Gas Meter Temperatures (T_m) and Average Orifice Pressure Drop (ΔH). See data sheet (Figure 5–3 of Method 5).

12.3 Dry Gas Volume, Volume of Water Vapor, and Moisture Content. Using data obtained in this test, calculate $V_{m(std)}$, $V_{w(std)}$, and B_{ws} according to the procedures outlined in Method 5, Sections 12.3 through 12.5.

12.4 Total Lead in Source Sample. For each source sample, correct the average absorbance for the contribution of the filter blank and the 0.1 N HNO_3 blank. Use the calibration curve and this corrected absorbance to determine the Pb concentration in the

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sample aspirated into the spectrophotometer. Calculate the total Pb content m_t (in µg) in the original source sample; correct for all the dilutions that were made to bring the Pb concentration of the sample into the linear range of the spectrophotometer.

12.5 Sample Lead Concentration. Calculate the Pb concentration of the sample using the following equation:

$$C_m = C_a \frac{A_m}{A_t - A_m} \qquad \text{Eq. 12-1}$$

12.6 Lead Concentration. Calculate the stack gas Pb concentration C_s using Equation 12–2:

$$C_s = K_3 \frac{m_t}{V_{m(std)}} \qquad \text{Eq. 12-2}$$

Where:

K_3 = 0.001 mg/µg for metric units.
= 1.54×10^{-5} gr/µg for English units

12.7 Stack Gas Velocity and Volumetric Flow Rate. Calculate the average stack gas velocity and volumetric flow rate using data obtained in this method and the equations in Sections 12.2 and 12.3 of Method 2.

12.8 Isokinetic Variation. Same as Method 5, Section 12.11.

13.0 Method Performance

13.1 Precision. The within-laboratory precision, as measured by the coefficient of variation, ranges from 0.2 to 9.5 percent relative to a run-mean concentration. These values were based on tests conducted at a gray iron foundry, a lead storage battery manufacturing plant, a secondary lead smelter, and a lead recovery furnace of an alkyl lead manufacturing plant. The concentrations encountered during these tests ranged from 0.61 to 123.3 mg Pb/m³.

13.2 Analytical Range. For a minimum analytical accuracy of ±10 percent, the lower limit of the range is 100 µg. The upper limit can be extended considerably by dilution.

13.3 Analytical Sensitivity. Typical sensitivities for a 1-percent change in absorption (0.0044 absorbance units) are 0.2 and 0.5 µg Pb/ml for the 217.0 and 283.3 nm lines, respectively.

14.0 Pollution Prevention. [Reserved]

15.0 Waste Management. [Reserved]

16.0 Alternative Procedures

16.1 Simultaneous Determination of Particulate and Lead Emissions. Method 5 may be used to simultaneously determine Pb provided: (1) acetone is used to remove particulate from the probe and inside of the filter holder as specified by Method 5, (2) 0.1 N HNO_3 is used in the impingers, (3) a glass fiber filter with a low Pb background is used, and (4) the entire train contents, including the impingers, are treated and analyzed for Pb as described in Sections 8.0 and 11.0 of this method.

16.2 Filter Location. A filter may be used between the third and fourth impingers provided the filter is included in the analysis for Pb.

16.3 In-Stack Filter. An in-stack filter may be used provided: (1) A glass-lined probe and at least two impingers, each containing 100 ml of 0.1 N HNO_3 after the in-stack filter, are used and (2) the probe and impinger contents are recovered and analyzed for Pb. Recover sample from the nozzle with acetone if a particulate analysis is to be made.

17.0 References

Same as Method 5, Section 17.0, References 2, 3, 4, 5, and 7, with the addition of the following:

1. Perkin Elmer Corporation. Analytical Methods for Atomic Absorption Spectrophotometry. Norwalk, Connecticut. September 1976.

2. American Society for Testing and Materials. Annual Book of ASTM Standards, Part 31: Water, Atmospheric Analysis. Philadelphia, PA 1974. p. 40–42.

3. Kelin, R., and C. Hach. Standard Additions—Uses and Limitations in Spectrophotometric Analysis. Amer. Lab. 9:21–27. 1977.

4. Mitchell, W.J., and M.R. Midgett. Determining Inorganic and Alkyl Lead Emissions from Stationary Sources. U.S. Environmental Protection Agency. Emission Monitoring and Support Laboratory. Research Triangle Park, NC. (Presented at National APCA Meeting, Houston. June 26, 1978).